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UNITED STATES
DEPARTMENT OF THE INTERIOR
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BUREAU OF MINES
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INFORMATION CIRCULAR

PREPARATION OF THIN SECTIONS OF COAL

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BY

R. THIESSEN, G. C. SPRUNK, AND H. J. O'DONNELL

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UNITED STATES DEPARTMENT OF THE INTERIOR - BUREAU OF MINES

PREPARATION OF THIN SECTIONS OF COAL[1]

By R. Thiessen[2], George C. Sprunk[3],
and H. J. O'Donnell[4]

INTRODUCTION

The technique of preparing thin sections of coal by the method developed at the Bureau of Mines was first described in 1920[5] and again in 1931[6]. The latter date marked the beginning of an extensive program having as one of its objectives the correlation of the microscopical analyses of various American coals with their gas-, coke-, and byproduct-making properties.

This systematic microscopical examination, which has been extended to approximately 50 coal columns, has effected a number of improvements and refinements in coal-sectioning technique and has evoked numerous requests for instructions in preparing thin sections of coal by the latest Bureau of Mines method. This Information Circular has been published in response to such requests.

For convenience, the report has been divided into four sections, which are presented in the following order and which give instructions for (1) taking a carbonization sample in the mine; (2) removing a column of coal from the mine for subsequent macroscopic examination and microscopic analysis; (3) preparing, for macroscopic study, relatively large polished blocks representing the entire column of coal; and (4) preparing thin sections of the column of coal for microscopic analysis.

CARBONIZATION SAMPLE

In the Bureau of Mines survey of gas-, coke-, and byproduct-making properties of American coals, the carbonization sample and the sample for microscopic examination are taken at the same place in the mine and in that part of it considered to be most representative of the coal. Because the two samples are related so closely in the Bureau of Mines work, it has been deemed advisable to describe the method for taking a carbonization sample.

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[1] The Bureau of Mines will welcome reprinting of this paper, provided the following footnote acknowledgment is used: "Reprinted from Bureau of Mines Information Circular 7021".

[2] Senior research chemist, Central Experiment Station, Bureau of Mines, Pittsburgh, Pa.

[3] Assistant coal microscopist, Central Experiment Station, Bureau of Mines, Pittsburgh, Pa.

[4] Junior laboratory helper, Central Experiment Station, Bureau of Mines, Pittsburgh, Pa.

[5] Thiessen, R., Structure in Paleozoic Bituminous Coals: Bull. 117, Bureau of Mines, 1920, pp. 10-13.

[6] Fieldner, A. C., Davis, J. D., Thiessen, R., Kester, E. B., and Selvig, W. A., Methods and Apparatus Used in Determining the Gas-, Coke-, and Byproduct-Making Properties of American Coals: Bull. 344, Bureau of Mines, 1931.

A freshly exposed place representative of the coal in the mine is selected as the sampling point, and the face is "squared up" so that there is no undue overhang of top coal. After determining, by calculation, the size of the block of solid coal that will yield a 4,000-pound carbonization sample, the kerf (see fig. 1A) is cut to the proper width and depth by an undercutting machine or other suitable device. The coal should be undercut as near the bottom as possible, but the cut should not extend into the bottom rock. The cuttings should be saved and loaded with the carbonization sample. After being undercut, the coal is shot down with a minimum of explosive placed in the drillhole X (see fig. 1A). All loose coal and overhang from the cut is then loaded for the carbonization sample.

COLUMN SAMPLE FOR MACROSCOPIC AND MICROSCOPIC EXAMINATION

The ideal sample for microscopical examination is a square pillar or column of coal cut perpendicular to the bedding plane and extending from the top to the bottom of the bed. The size of this pillar will depend on the scope of the work, and the method of cutting it out will depend on the nature of the coal and the type of equipment available.

Laying Off the Column Sample on the Mine Face

After the carbonization sample has been removed, two faces are exposed (see fig. 1A) The column from which the samples for macroscopic and microscopic examination will be prepared is marked off on these faces by drawing two lines parallel to the long axis of the column, as shown in figure 1B. If the column is to be removed in a single piece, it should measure not less than 9 inches on each of the four sides. Furthermore, it should measure not more than 12 or 13 inches on a side, or it will be too heavy for easy handling. The top and bottom of the column should be designated by affixing adhesive-tape markers bearing the words "top" and "bottom". Additional adhesive-tape markers should be placed on both faces of the column and the distance from the top of the bed to the marker recorded thereon (see fig. 1B), so that if the column should break, the pieces may be assembled correctly.

Removing Column Sample from Bed

After the column sample has been marked off, a series of closely spaced holes is drilled along the line drawn on each exposed face (see fig. 1B). These holes are drilled about 16 inches deep, and the intervening solid coal is removed with a pick, leaving the column free on all sides and fixed at the top and bottom. The column is then released at the top either by cutting into the roof with a pick or by drilling and breaking the roof, if a compressor and air rock drills are available. In some cases the column may be released by making a cut above the coal with a crosscut saw.

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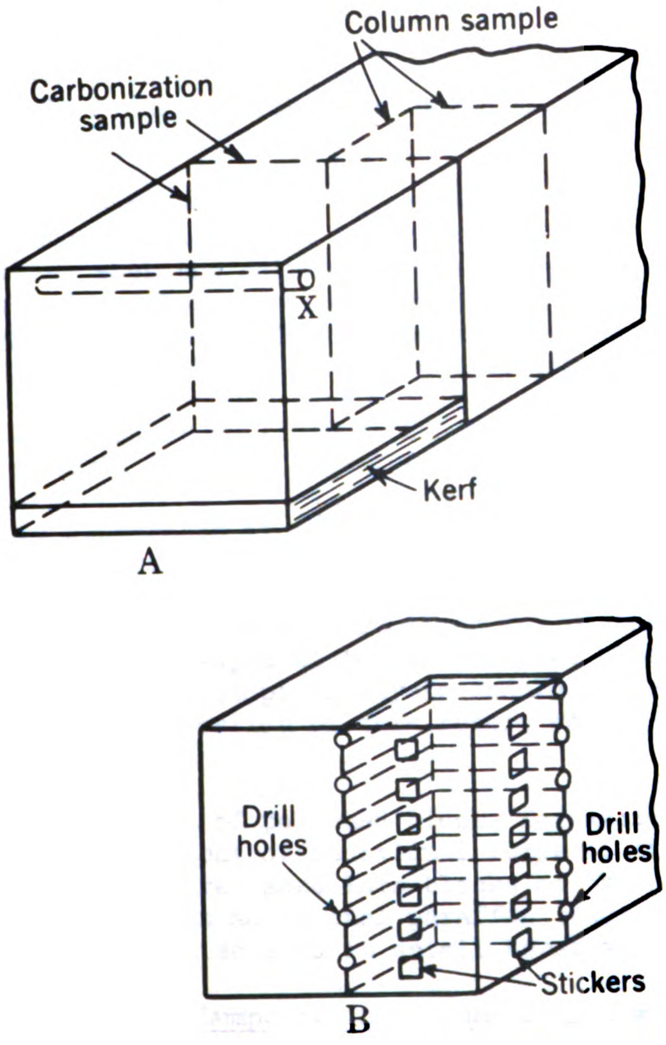

Figure 1.- A, Method used by the Bureau of Mines in obtaining a column sample adjacent to a 4,000-pound carbonization sample. After undercutting, the carbonization sample is shot down at point X. The undercuttings, all loose coal, and the overhang or top coal are loaded for the carbonization sample. B, Sketch showing method of freeing column by drilling a series of closely spaced holes on the two exposed faces. The coal left between the holes is removed with a pick, so that the column is free except at the top and bottom. Positions from the top of the bed are marked on the adhesive-tape stickers.

After the column has been freed at the top, a trench 3 to 4 inches deep should be cut into the floor and the column wedged free of the bottom. The wedges should be driven into the bottom rock and not between the coal and bottom rock. The column is now free on all sides, top, and bottom, and, after boards are wired as tightly as possible on all four sides, it is ready to be removed. It is important that the boards be wired as tightly as possible, first, to add strength to the column of coal and, second, to retain the pieces of the column in the event it breaks, which frequently happens in handling during removal.

After the boards have been wired on securely, the trench at the bottom should be extended about 1½ to 2 feet in front of the column, to provide sufficient room for turning when the column is removed from the bed. The column may now be slipped forward and lowered to a horizontal position and packed securely to prevent breakage in shipment. Packing materials such as excelsior, wood shavings, straw, hay, burlap bags, canvas, and brattice cloth are satisfactory.

PREPARATION OF POLISHED BLOCKS TO REPRESENT THE MACROSCOPIC APPEARANCE OF THE ENTIRE COLUMN SAMPLE

The macroscopic appearance of the entire coal column may be studied by preparing a series of relatively large polished blocks, each of which represents a section of the original column. The macroscopic appearance and the microscopic analysis may be correlated if the thin sections for microscopic examination are made from slices immediately adjacent to the polished blocks. The exact position of the thin sections for microscopic analysis should be recorded on the polished blocks as described in that portion of this report which gives instructions for preparing thin sections.

Briefly, the preparation of polished blocks for macroscopic examination involves a preliminary treatment of individual lumps of the column sample with paraffin, after which these are sawed into blocks of the proper dimensions. The sawed blocks are then given an additional paraffin treatment, following which they are polished. Each step is discussed in detail hereinafter.

Treating Individual Lumps of the Column Sample with Paraffin

It is frequently difficult to prepare a polished block of the desired size and shape because of the friability of the coal. To overcome this difficulty, friable coals should be soaked over night in a paraffin bath maintained at 110°C. In some cases, shattering during sawing may be prevented merely by painting the surface of the coal with melted paraffin.

Sawing Out the Block to be Used for Macroscopic Examination

The individual blocks of the column sample are cut with a 12-inch Norton bakelite bonded Crystolon grinding wheel, 1/8-inch thick, which is run at a speed of approximately 1,500 revolutions per minute. No water or abrasive is used. A Norton wheel of grain size 3736 and grade Q4T-2 has proven satisfactory. The grain size should not be too small, or the wheel will tend to form a thin, gummed layer on the cutting edge.

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Before sawing out the block for polishing, the directions in which the lump may split should be considered. Horizontal fractures occur in the bedding plane, which lies at right angles to the long axis of the column. In addition to the horizontal breaks, there are generally two vertical cleavage systems. One of these systems is usually more pronounced than the other and consists of definite vertical cleavages parallel to each other, along which the coal breaks fairly easily, generally with a more or less straight break called the face-side cleavage. The other vertical system is less marked or barely exists and is roughly at right angles to the plane of the face-side cleavage. Coal breaks less easily and generally more or less irregularly along the planes in this cleavage system termed the butt end.

From the foregoing, it is evident that coal tends to break in the form of parallelopipeds; the two horizontal cleavage surfaces tend to parallel each other; the face surfaces perpendicular to the horizontal planes also tend to parallel each other; and the butt ends are generally irregular but tend to be perpendicular to the horizontal cleavage surfaces.

Experience has shown that the butt-end surfaces are the most suitable for polishing. Accordingly, the block is cut to a 3- to 4-inch width on the butt surface and a 2-inch width on the face surface. The length of the blocks varies from 4 to 10 inches and usually is determined by the horizontal fractures or thin fusain bands in the column sample.

Cutting Slice for Thin Sections

Before proceeding with the treatment of the block for macroscopic examination, a slice, from which the thin sections for microscopic analysis will be prepared, should be cut off. Figure 2 illustrates the butt-end surface of a sawed block on which the cutting line for the slice for thin sections has been marked. The cut should be made through the white line and perpendicular to the butt surface, as shown in figure 3. The preparation of the thin sections from the slice is the subject of a later section of this report.

Final Treatment of Sawed Block With Paraffin

After cutting off the slice for thin sections, the blocks should be heated in a bath of paraffin at 105° to 110° C. for at least 24 hours. The paraffin acts as an adhesive and reduces friability.

Polishing Sawed Block for Macroscopic Examination

The butt end of the sawed block should then be ground to a plane surface on a series of three plate-glass plates. (Ordinary window glass is not suitable.) A coarse abrasive (no. 120 carborundum powder) is used on the first plate, a medium abrasive (no. F carborundum) on the second plate, and a very fine abrasive (no. 600 carborundum) is used on the third plate. A circular or figure-eight motion is preferable for polishing, and since a perfect plane surface must be maintained on the plates, the whole surface of the glass plate should be used for grinding so that any unevenness or grooving will be avoided. After moistening the plates with water, the abrasive, either dry or as a paste, may be put on

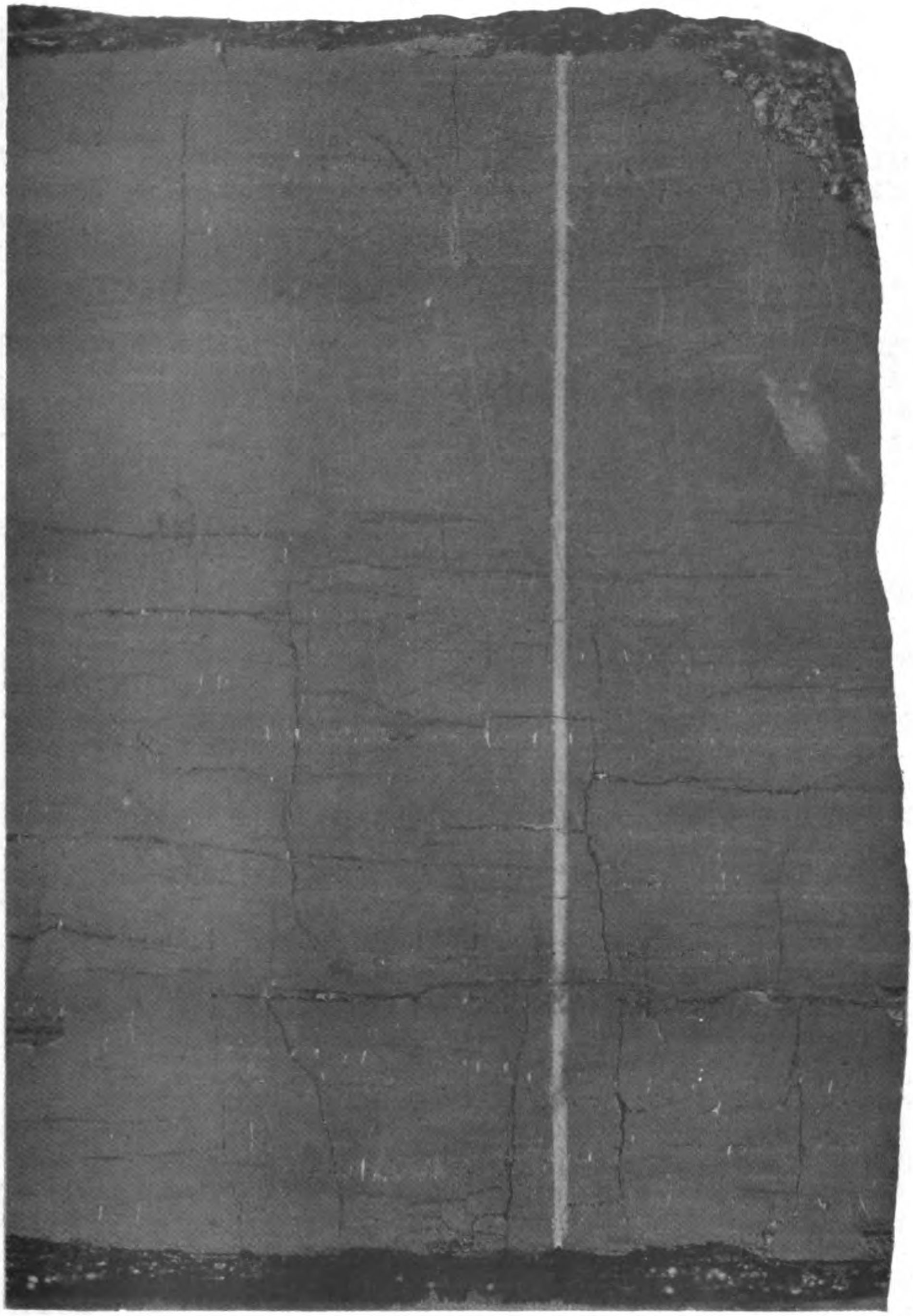

Figure 2.- First stage in polishing a block of coal. The butt-end surface of a block is sawed to the proper dimensions (3 to 4 inches wide). The slice for thin sections is cut perpendicular to the butt-surface through the white line.

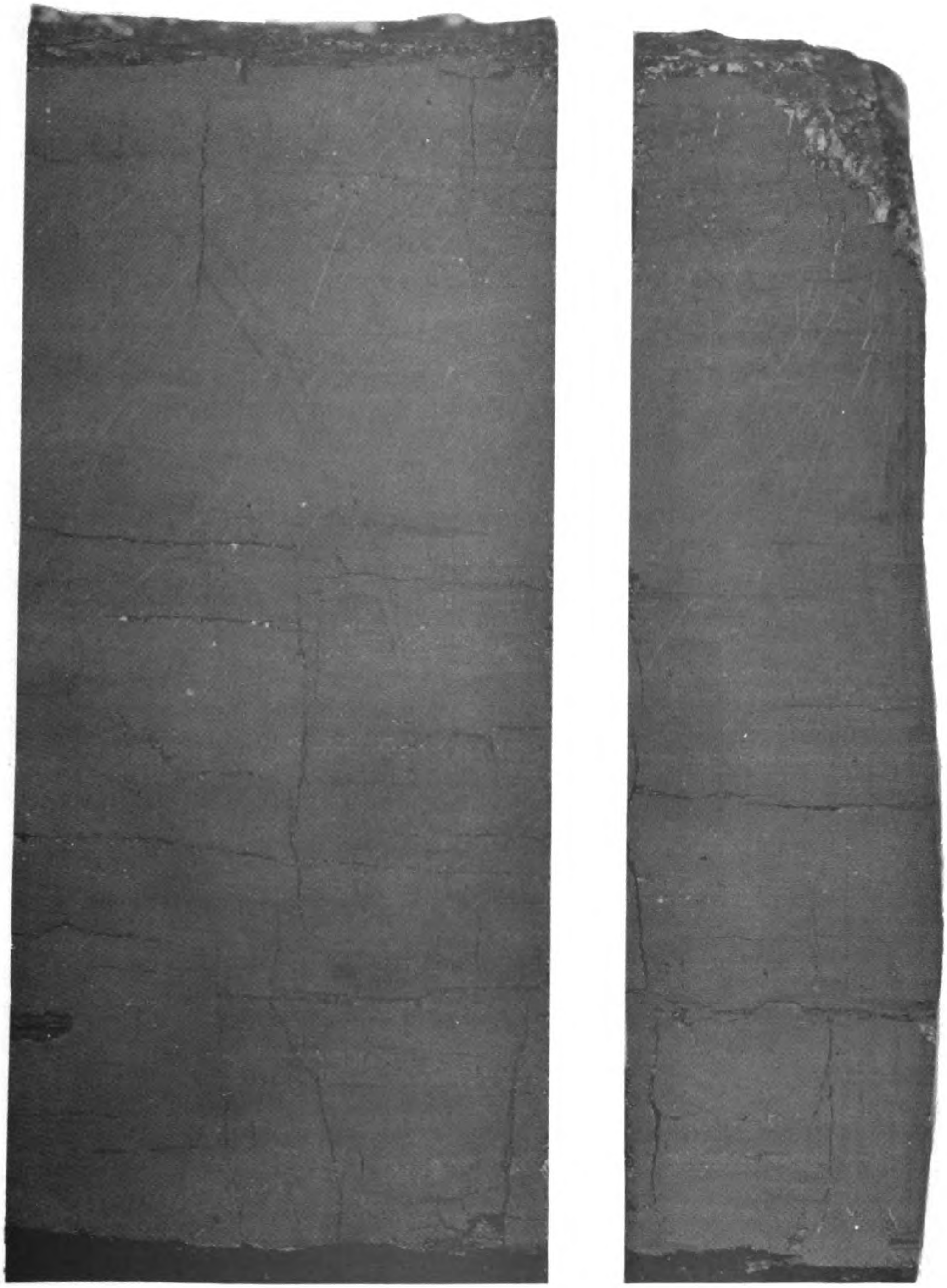

Figure 3.- The slice, cut adjacent to the block, is used for making the thin sections.

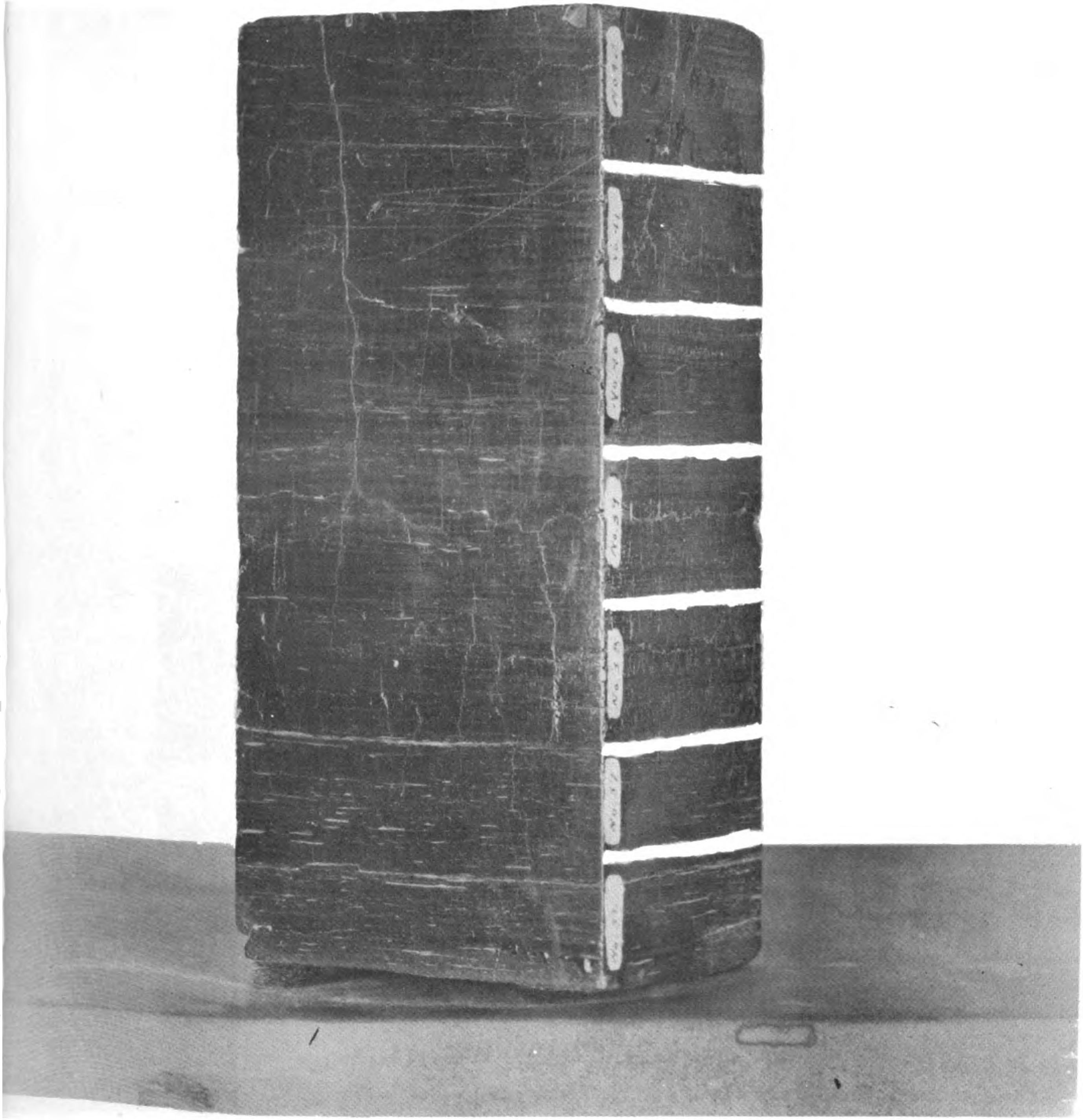

Figure 4.- Manner in which the exact position of the thin sections is recorded on the side of the polished block.

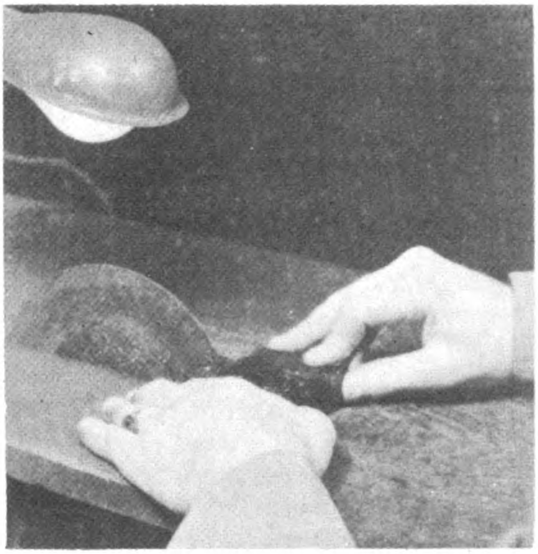

Figure 5.- A cube of coal is cut for the thin section with a small Norton grinding wheel.

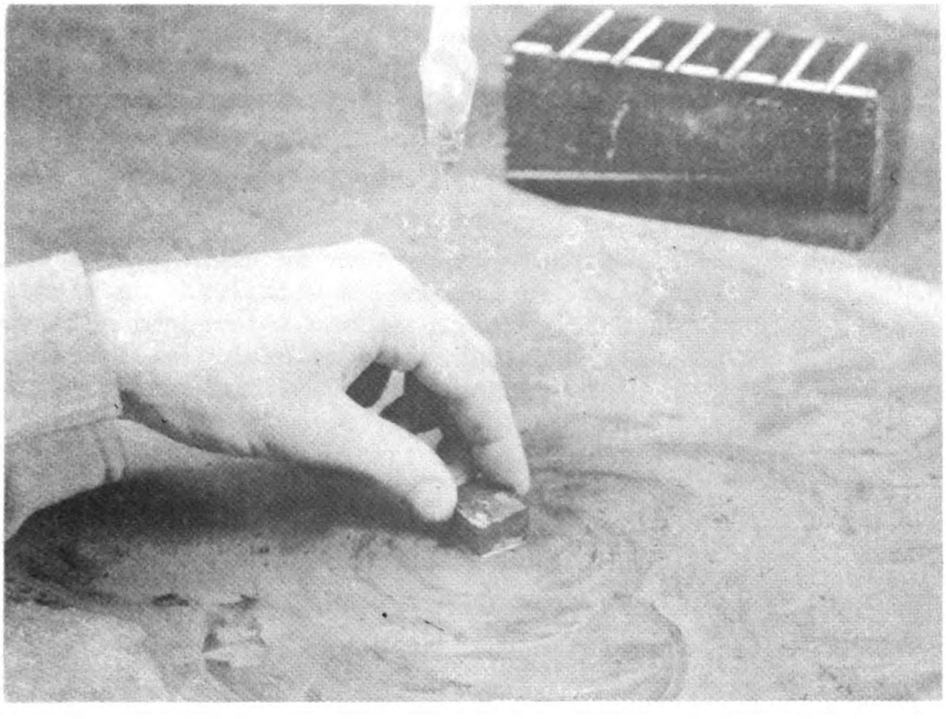

Figure 6.- Method of grinding block to a plane surface, first on a plate-glass plate with coarse carborundum (no. 120), next with a finer-grained abrasive, and then with the finest-grained abrasive possible.

with a spatula. Each grinding is considered finished when all scratches or roughness caused by the previous grinding have been removed. The abrasive should be removed from the block by washing thoroughly after each grinding stage.

The polishing is completed on a Selvyt cloth (a British cotton cloth with a very fine nap used by jewelers in polishing) with a paste of slaked lime. A Selvyt cloth approximately 14 inches square is fastened with thumb tacks around the edge of a plane-surface board, so that the entire plane-surface is available for polishing. A supply of calcium oxide may be slaked and kept in the form of a paste in a well stoppered bottle, for use as required. The paste should be passed through a 200-mesh sieve by brushing to remove any grit; and particular care must be taken to keep it away from the air to prevent the formation of calcium carbonate.

The final polish is imparted by rubbing the prepared face of the block with a straight, forward pull over the Selvyt cloth, which has been moistened previously and covered to a considerable extent with slaked-lime paste. A straight, forward pull across the bedding plane defines the layering and boundaries of the constituents more sharply than a circular or figure-eight motion. With most coals, a certain maximum gloss will be obtained, after which a dull surface will result if the specimen is overpolished.

PREPARATION OF THIN SECTIONS FOR MICROSCOPIC EXAMINATION

The thin sections for microscopic examination are prepared from the slice of coal that was cut adjacent to the polished block. The cutting of this slice has been described already in this report.

Treating Slice of Coal with Paraffin

The slice of coal for thin sectioning (see figs. 2 and 3) should be heated at least 24 hours in paraffin at about 105° to 110° C. Heating drives off the moisture and gases that would be evolved and that would remain as tiny bubbles under the section when the finished specimen is heated and cemented to the object slide. The paraffin penetrates the specimen, particularly crevices, and renders it more compact and less liable to break during further manipulation. After the 24-hour period, the paraffin bath is permitted to cool to near the solidification point, after which the slice of coal is removed and plunged into cold water. In this way, the paraffin will be retained within the coal slice.

Sawing the Small Block to be Cemented on Object Slide

When the slice has cooled thoroughly, it is marked off into divisions approximately 3/4 to 1 inch wide. Corresponding divisions are marked on the side of the original large polished block and each division is given a thin-section number, as illustrated in figure 4. In every instance, a cube of coal of the same dimension as the width of the division is cut out for the thin section by means of a small Norton grinding wheel, 8 by 1/16 inches (see fig. 5).

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Grinding and Polishing the Small Block to be Cemented on Object Slide

One side of the small block must be polished before it is cemented on the object slide preparatory to making the thin section. For this purpose, the butt-end surface is chosen again, chiefly so that the polished surfaces of the small blocks will coincide with the polished surfaces of the larger blocks. Thus, it is possible to compare the microscopic appearance of the thin sections with the macroscopic appearance of a contiguous cross section of the bed.

The butt-end surface of the small block should be ground to a plane surface first on a plate-glass plate with coarse carborundum, next with a medium grained abrasive, and finally with a very fine-grained abrasive. These three grinding operations have been explained previously in the instructions for grinding the larger blocks. The last of these grinding stages is illustrated in figure 6. As previously stated, each grinding may be considered finished when all scratche and roughness caused by the previous grinding have been removed. Furthermore, the abrasive should be removed from the block by washing it thoroughly after eac grinding.

The surface of the coal is then rubbed to a higher polish with a long forward stroke over a very fine-grained, yellow, Belgian hone lubricated by means of small stream of water (see fig. 7). After this, the block is given the finis ing polish on the Selvyt cloth, as shown in figure 8. This step is the same as that for the larger blocks. However, as the samples are relatively small, only a few rubs are necessary to give the surface the required polish.

Cementing the Small Blocks on Object Slide

Cementing the blocks on object slides requires prepared cement and a warming oven. Cement made of Canada balsam and marine glue has been used successful in the Pittsburgh laboratory for some time.

The cement is prepared by heating and stirring together at 105° to 110° C. about three parts of Canada balsam dissolved in xylene and two parts of refined marine glue. This procedure is continued until the mixture, when cooled, is hard yet tough. It must not be too hard nor too brittle, yet it must be hard enough not to give way under the section during grinding. The correct consisten must be determined by trial. In general, the hardness is correct when at room temperature the cement can barely be indented with the thumb nail. When the cor rect consistency has been reached, the prepared cement will remain a long time without change and may thus be kept on hand for immediate use.

Any ordinary warming oven heated by gas or electricity that can be maintai at 105° to 110° C., is suitable. Preferably, it should have a shelf in the cent able to support a considerable weight and withstand some pressure.

In cementing the small block on the slide, first a small amount of the cem enough to spread a thin film over the surface of the block, is placed on an ordinary, medium-thick, 1- by 3-inch, white-glass object slide. The slide is th

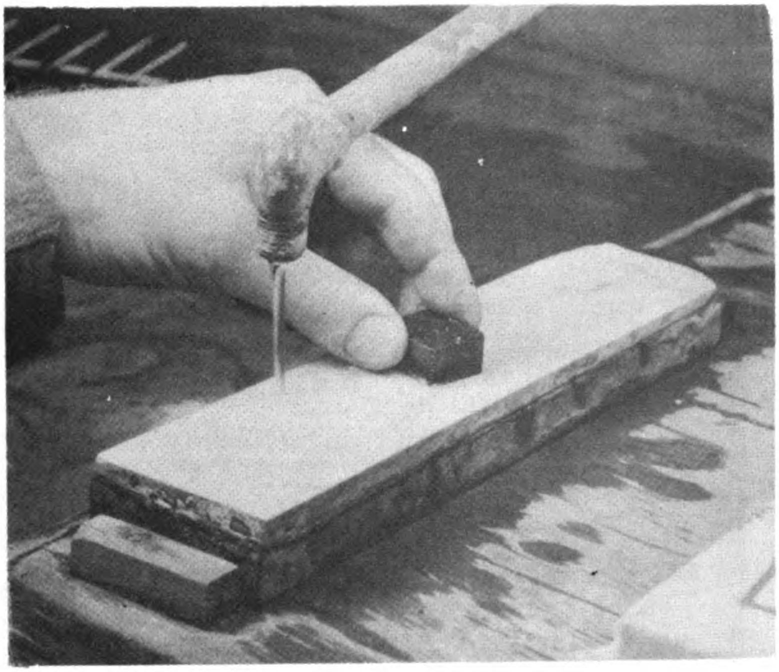

Figure 7.- Rubbing the plane surface of the block to a higher finish on a very fine-grained yellow Belgian hone lubricated by a small stream of water.

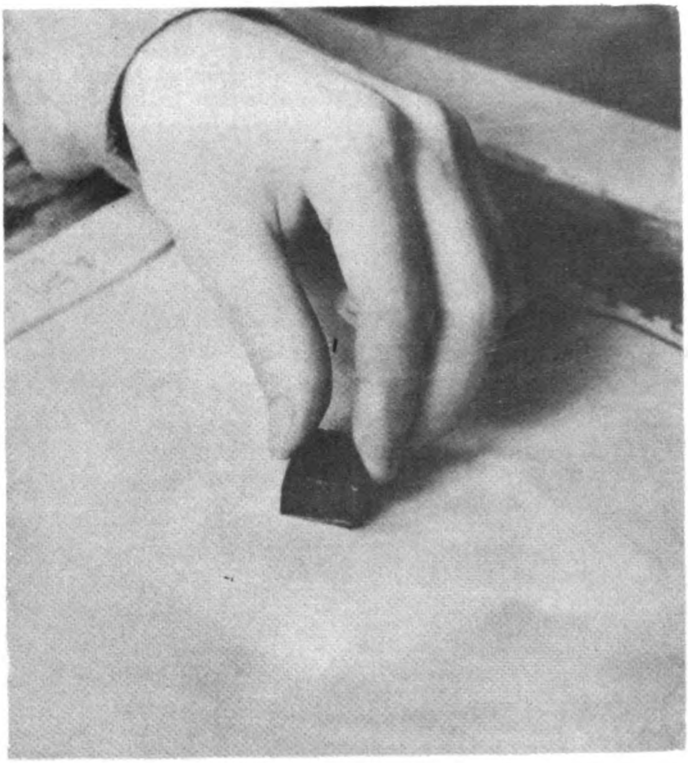

Figure 8.- Polishing the block on the Selvyt cloth with slaked calcium oxide as the polishing medium.

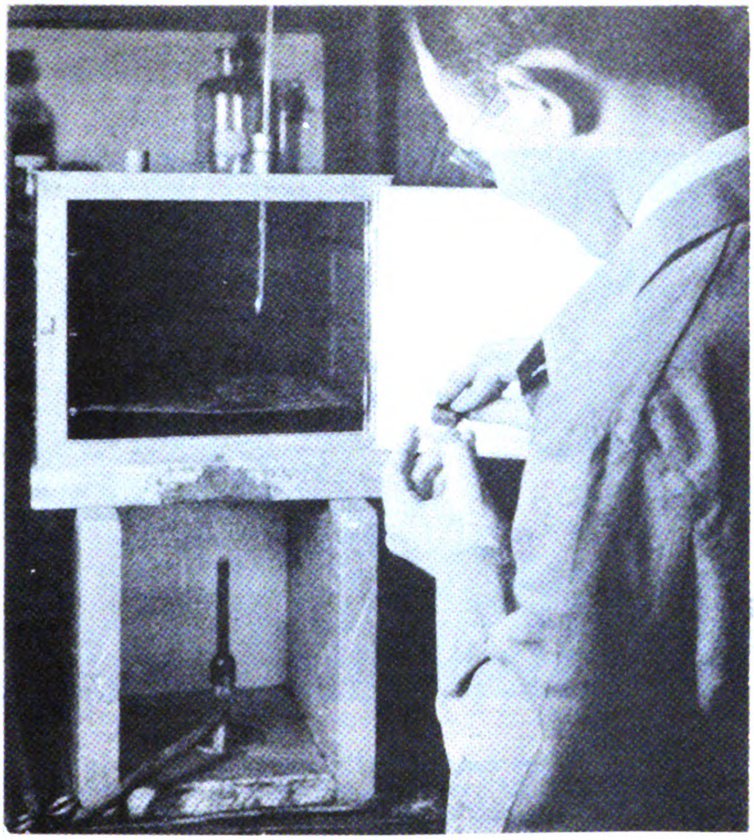

Figure 9.- Cementing the polished block to a glass object slide. It is absolutely necessary to have a thin, even film of cement between the specimen and the object slide or the succeeding operations will fail.

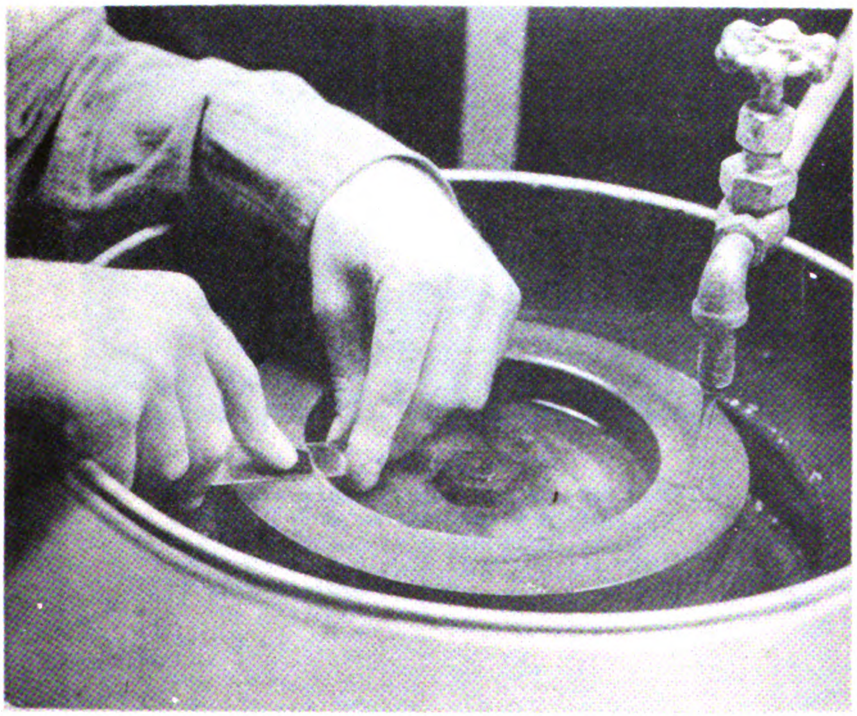

Figure 10.- Grinding the section as thin as possible on the rim of a fine-grained, 9-inch carborundum wheel.

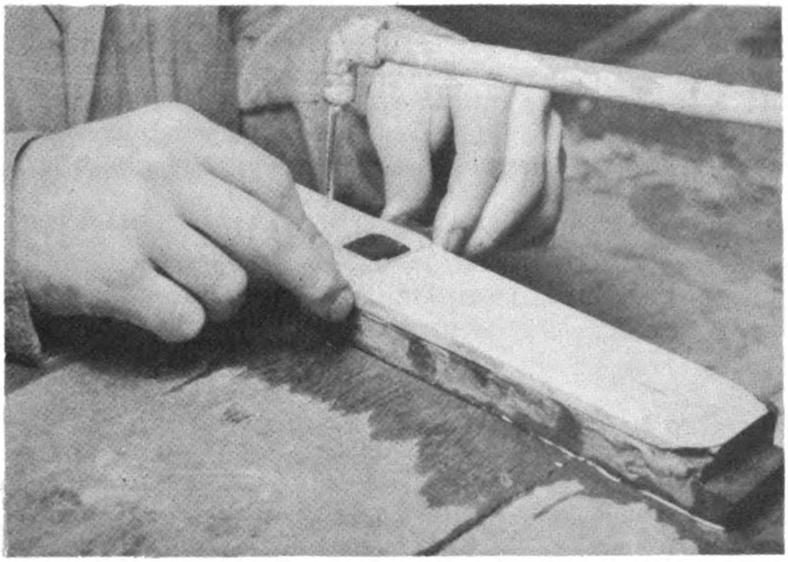

Figure 11.- On a flat, fine-grained razor hone lubricated by a stream of water, the thin section is ground to the proper thinness. If there is any tendency for the thin section to be uneven, differential pressure should be applied by the fingers during the rubbing process.

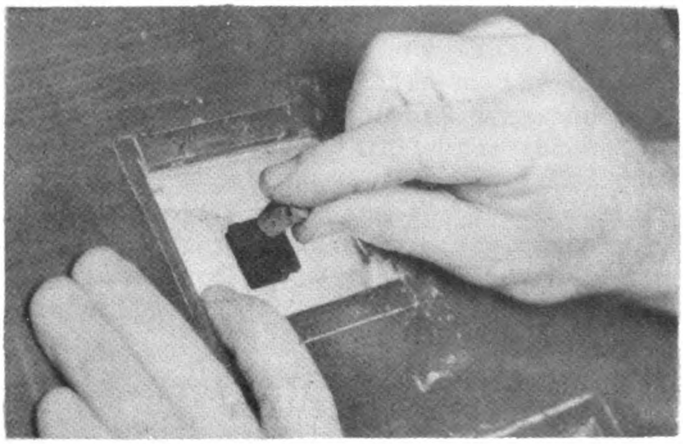

Figure 12.- Final stage in preparing thin section. The section, held against a lighted window, is polished by using a softened cork dipped in Goddard's nonmercurial plate powder.

placed on the shelf of the oven. When the cement is warm enough to flow freely, the polished surface of the block is held near the flame of a Bunsen burner just long enough to heat the surface without heating the block through. Then the heated surface of the specimen is pressed quickly into the melted cement; the slide with the block is removed from the oven, and the block is pressed firmly to the slide, as in figure 9. If the proper amount of pressure is applied, any bubbles that may form will be forced to the edge of the specimen, and at the same time a thin film of cement will remain between the slide and the block. It is absolutely necessary to have a thin, even film of cement between the specimen and the object slide, or subsequent operations will fail.

Grinding Cemented Block

When the cemented block is cool and the glue has hardened, the larger part of the coal is sawed off with a thin Norton wheel, leaving about 1/4 inch of coal on the slide.

In the next step, the section is ground first on a coarse-grained lapidary's wheel and then on a fine-grained 9-inch wheel with a rim, such as is illustrated in figure 10. Either wheel can be fed with size F carborundum and a small stream of water. Although no attempt is made to finish the section on the wheel, it is ground as thin as possible without injury to it. Too close grinding on the wheel will shatter the thin section.

After the superfluous cement is trimmed around the edges of the section with a safety-razor blade, the work is transferred to a very fine-grained, yellow, Belgian hone. A hone 25 cm's long and of the finest grain possible has been found to be best for grinding the sections to the required thinness. The hone is fixed on a pedestal sloping slightly away from the worker, and a thin stream of water is played upon it (see fig. 11). The specimen is rubbed by using long forward strokes and slight pressure. If there is any tendency for the section to be uneven, differential pressure should be applied by the fingers during the rubbing procedure. The hone can be cleaned and planed occasionally by actually grinding it on a wet, plate-glass plate with no. 600 carborundum. As long as the section remains relatively thick, no great care is required and considerable pressure may be applied; but when the sections approach the correct thinness, great care must be taken and the pressure must be reduced proportionately. When the specimen presents a uniformly illuminated field with sufficient light transmitted through it to bring out the constituents, the grinding is considered complete.

Polishing the Section

Figure 12 shows the method used in the polishing operation, which is the final stage in the preparation of thin sections. The section is held against a lighted window and the polishing is accomplished by using a softened cork (previously soaked in water) and a small amount of Goddard's non-mercurial plate powder (a polishing powder made by J. Goddard & Sons, Ltd., Leicester, England). With this powder, it is possible actually to rub down any thick areas of the section.

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The slide with the section is cleaned carefully with xylene and covered
with a no. 1 cover glass over a drop of Canada balsam or cedar oil; or the cove
glass may be put on temporarily with a drop of glycerin. Finally, the section
is labeled and filed for microscopic examination.

Usually, about 75 thin sections are prepared from a 5-foot coal bed, and
approximately 2 weeks are required to prepare the sections with accompanying
polished blocks. A single thin section can be made in about 15 minutes if the
coal does not require preliminary treatment.

One of the more frequent inquiries concerns the range of coals that can b
sectioned. It is well known that as the coal increases in rank, it becomes increasingly difficult to make sections thin enough to transmit light, and, final
a limit is reached at which light will not be transmitted even through the thin
nest sections. However, all coals from the lignites to the upper part of the
low-volatile bituminous group can be sectioned successfully. Coals having approximately 17 percent volatile matter (dry, mineral-matter-free basis) yield
sections that will permit a study of the type of material of which they are co
posed, although these sections are not of high quality, for such coals are usua
soft and extremely friable. They can be improved by impregnating them with bak
lite in the following manner:

The specimens for mounting on the object slides are put in a dish, covered
with Westinghouse no. 411 bakelite varnish, and left in a vacuum desiccator for
12 hours. The blocks are then removed and left in a 70° C. oven until the bake
lite has hardened thoroughly, after which the blocks are sectioned in the usual
manner.